Close-Proximity Electromagnetic Carbonization (CPEC)

(*Low Temperature Carbonization, LTC*)

Mat122

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Partner:
4X Technologies, LLC.

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Overview

Timeline

- Project Start: 10/1/15
- Project End: 9/30/20*
- Progress: ca. 75%

*This project had two extensions:
- FY19 at no additional cost
- FY20 with additional costs

Budget

Initial budget planning
- FY16 – FY19: $4.5M

Effective budget:
- Funding received in FY16: $1.5M
- Funding for FY17: $1.35M (10% cut)
- Funding for FY18: $1.5M
- Funding for FY19: $0 (ext. at no add. costs)
- Funding for FY20: $1.0M

Barriers

- Barriers addressed
  - Cost: A goal of this project is to reduce energy consumption in the carbon fiber conversion process and therefore total carbon fiber cost.
  - Inadequate supply base: Another goal of this project is to reduce the required processing time for carbonization and therefore increase overall throughput.

2017 U.S. DRIVE MTT Roadmap Report, section 4

Partners

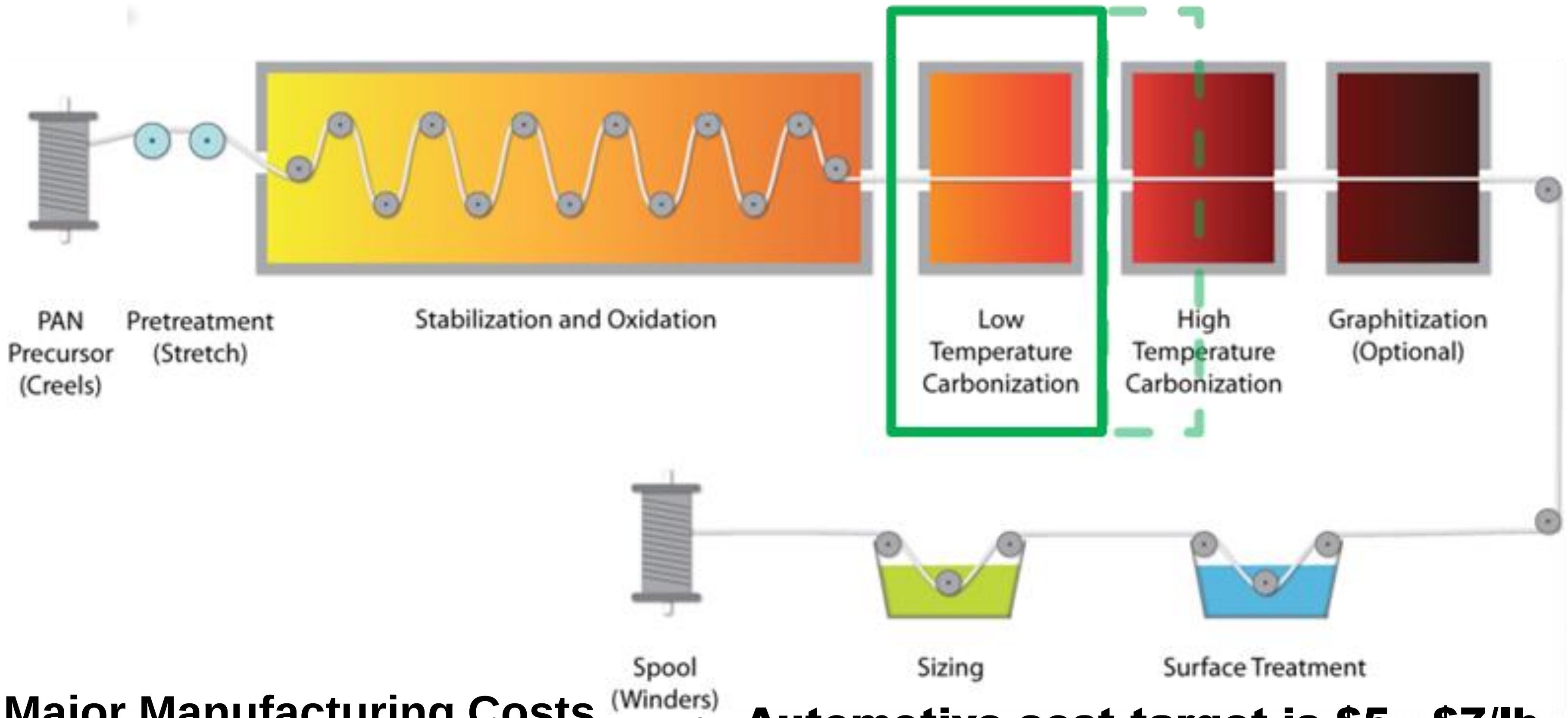
- Project lead: ORNL
- Partner: 4X Technologies

(formerly RMX Technologies)

Relevance

- Project title:
  Close Proximity Electromagnetic Carbonization (CPEC):
  - Low temperature carbonization process (LTC)
  - Relies on dielectric heating (no convection)
  - Faster and more efficient that conventional
  - At atmospheric pressure.
- Project Goals:
  - Reduce unit energy consumption of LTC stage (kWh/kg) by ca. 50% (ca. 5% of the cost reduction on the CF overall manufacturing process).
  - Produce equal or better quality carbon fiber.
  - Scale the technology to a nameplate capacity of 1 annual metric ton and demonstrate by project end date (in progress).

Approach (conventional PAN processing)



Major Manufacturing Costs	
Precursor	43%
Oxidative stabilization	18%
Carbonization	13%
Graphitization	15%
Other	11%

- **Automotive cost target is $5 - $7/lb**
- **Tensile property requirements are 250 ksi, 25 Msi, 1% ultimate strain**
- **ORNL is developing major technological breakthroughs for major cost elements**

Approach (CPEC)

- Conventional furnaces consume significant energy heating large volumes of inert gas surrounding the fiber.
- If thermal energy could be directly coupled from an energy source to the fiber, tremendous energy savings could be realized.
- This project uses electromagnetic coupling to directly heat the fiber – not the surroundings (hardware, gas, etc.).
- Dielectric/Maxwell-Wagner heating mechanisms are utilized.

$$P_v = 2\pi f |E|^2 \varepsilon_0 \varepsilon' tan\delta$$

  - P_v volumetric power transferred to the material.
  - ε' is the relative dielectric constant.
  - ε_0 is permittivity of free space, 8.85418782 x 10^{-12} F/m.
  - $|E|$ is the magnitude of the local electric field intensity (V/m).
  - $tan\delta$ is the loss tangent of the material.
  - f is the operational frequency.

FY20 Milestones*

Date	Milestone	Status
Dec 31, 2019	**M12:** CEM** iterative work completed, hardware modification known.	**Completed: 3/2019**
March 31, 2019	**M13:** Hardware modification completed	**Completed: Apr. 27, 2020**
June 30, 2020	**M14:** 4 tows processed (final CF: strength = 250 ksi, Modulus = 25 Msi).	**In progress**
Aug. 31, 2020	**M15:** 4 tows processed (final CF: strength = 250 ksi, Modulus = 25 Msi, residence time < 1min).	
Sept. 30, 2020	**M16:** Unit energy consumption of LTC by ca. 50% when compared to conventional LTC	

** Due to technical issue, and schedule adjustments, the milestone list was adjusted with the agreement of the DoE.*
A first project extension was approved until March 31, 2019.
A second extension was approved until September 30, 2019.

*** CEM : Computational Electromagnetic Modeling*

Historical development

- Project flow:

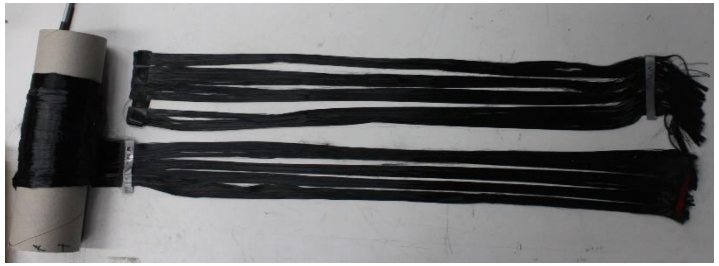
Material measurement → Computational Electromagnetic Modeling [CEM] (CPEC-2V) → Prototype design and build (CPEC-3) → Prototype tested → Prototype altered → Fiber production, Material characterization → CEM for CPEC-4 → Construction of CPEC-4 → CPEC-4 Commission and alteration → Traces of carbonization detected; Equipment failure → Upgrade: 2 new configurations → Currently testing one new configuration

(Comsol vs. CST)

Basic idea/confirmation (with CPEC-1, 2014-2015)

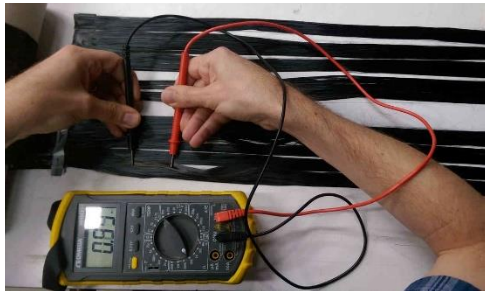
- Main achievements:
  - Material measurement/data acquisition (FY16)
  - CPEC-3 (FY17): demonstration of feasibility on batch or continuous process (one tow of 24k)
  - CPEC-4: in process of upgrade and test of two configurations.

CPEC-4 converted a section of fiber ...but part of the cavity failed (Sept 11, 2019)



Batch of 8 tows (50k) exposed for 5min in CPEC-4

A section of 8in long of carbonized fiber has was processed



The fiber became conductive: evidence of effective carbonization

Evidence of carbonization

Part of the processing cavity is damaged.

Some parts need modification

Export control: no picture of the setup can be showed

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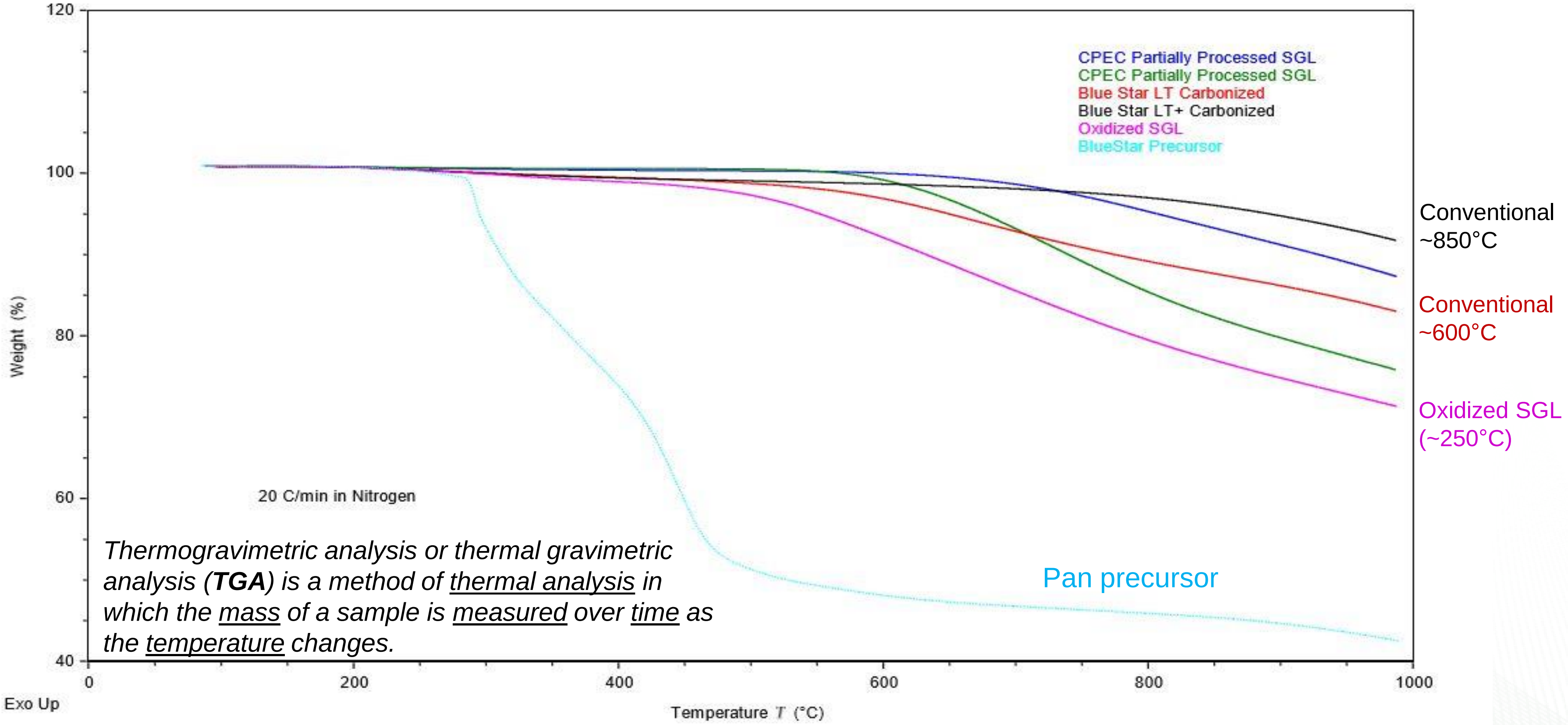
CPEC-4 processed fiber analysis (Sept. 2019)
TGA Results



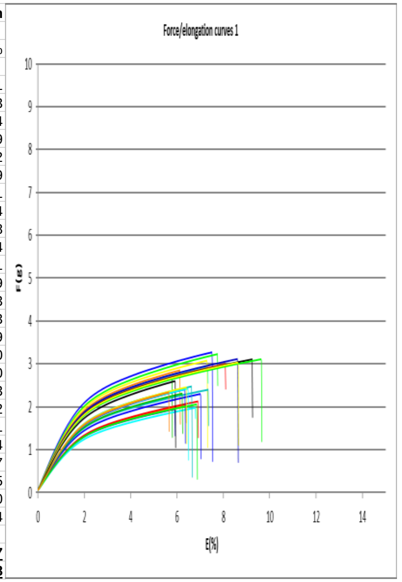
*Thermogravimetric analysis or thermal gravimetric analysis (**TGA**) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes.*

<u>Conventional:</u> the sample of interest has been carbonized using conventional process (no CPEC)

CPEC-4 processed fiber analysis (Sept. 2019)
Mechanical Analysis of LTC fiber

OPF processed feedstock fiber (control)

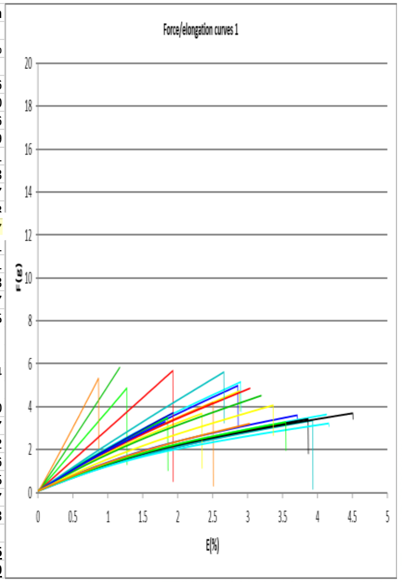
Specimen	Break Stress	Diameter	Modulus	Strain
Color	Ksi	µm	Mpsi	%
	38.90	12.35	1.53	7.51
	39.57	11.58	1.54	7.33
	40.55	12.01	1.55	7.74
	39.49	11.88	1.54	7.29
	37.93	11.67	1.56	6.12
	41.06	11.50	1.55	8.09
	42.42	11.53	1.57	8.61
	40.85	10.31	1.58	7.34
	38.01	11.26	1.59	5.78
	42.88	11.46	1.55	9.24
	41.17	10.06	1.61	7.01
	39.06	9.58	1.52	6.79
	43.84	11.34	1.56	9.63
	42.62	11.36	1.57	8.63
	40.26	9.59	1.60	6.39
	40.86	9.71	1.59	6.90
	38.62	9.56	1.59	5.90
	38.71	10.76	1.55	6.63
	39.37	10.31	1.60	6.22
	39.17	10.97	1.62	5.91
	40.32	10.40	1.63	6.34
	39.86	10.50	1.60	6.47
	40.47	9.60	1.59	6.85
	39.93	10.56	1.61	6.40
	38.80	10.45	1.63	5.64
	40.19	**10.81**	**1.58**	**7.07**
	1.53	**0.85**	**0.03**	**1.08**



Tensile: 40 ksi
Modulus: 1.6 Msi
Strain: 7.1 %

LTC fiber with CPEC-4 (carbonized region)

Specimen	Break Stress	Diameter	Modulus	Strain
Color	Ksi	µm	Mpsi	%
	84.00	10.37	3.35	2.86
	82.13	10.67	3.29	2.90
	55.72	10.65	3.23	1.86
	78.25	10.48	3.17	2.89
	69.27	10.48	3.17	2.51
	81.12	10.42	3.16	3.03
	66.39	9.44	2.48	3.67
	65.43	9.65	2.41	3.93
	126.41	9.15	10.64	1.17
	63.37	10.28	2.23	4.51
	61.85	10.28	2.37	3.71
	64.79	10.09	2.34	4.13
	92.04	9.80	7.14	1.27
	57.61	10.77	2.83	2.35
	128.93	8.66	14.71	0.87
	106.41	9.84	5.44	1.93
	69.79	9.83	3.76	1.94
	101.54	10.02	4.00	2.66
	71.15	10.73	2.74	3.20
	61.76	10.04	2.33	3.87
	56.36	10.30	3.30	1.82
	59.53	9.91	2.21	4.16
	57.08	10.22	2.28	3.55
	72.21	10.11	2.73	3.37
	57.61	10.05	2.47	3.03
	75.63	**10.09**	**3.91**	**2.85**
	20.84	**0.50**	**2.92**	**1.00**



Tensile: 76 ksi
Modulus: 3.9 Msi
Strain: 2.9 %

The large deviation of the LTC fiber is due to the probing method and the difficulty of separating processed and unprocessed fiber from a bundle.

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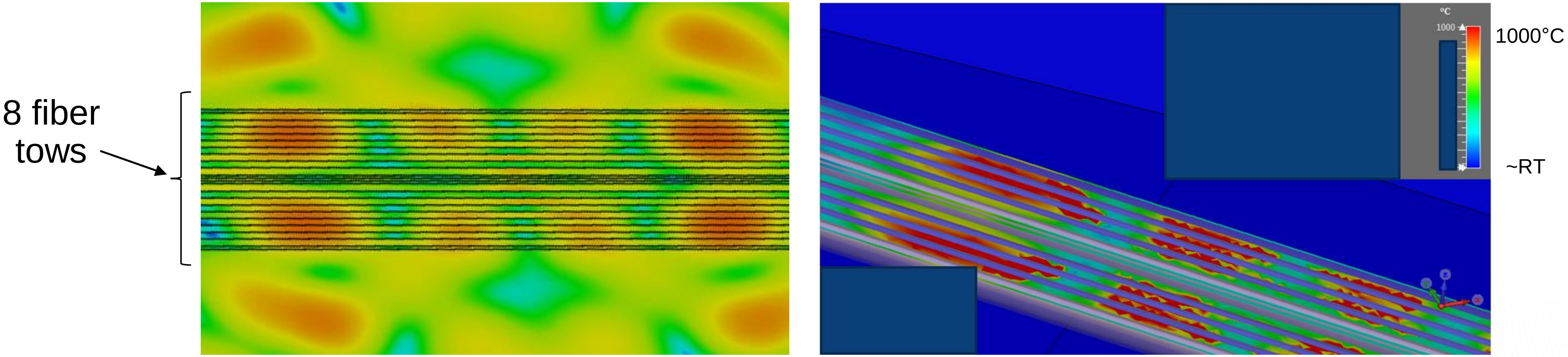
Approaches selected

- <u>CEM validation:</u>
  - Unlike with CPEC-3, the results with the CEM (CST) are not confirmed by the experience with CPEC-4
- <u>After multiple tests with CPEC-4:</u>
  - Several technical/engineering issues were identified
  - As is, the system did not match with expectation
  - Equipment component failure (Sept. 2019). Possible causes:
    - Material used
    - Geometry of the configuration
    - Some initial assumptions used in the CEM

→ NEED TO REVISIT THE MODEL in order to:
- **Improve the current configuration**
- **Explore new configurations**

Exploration of new configuration (Dec. 2019)

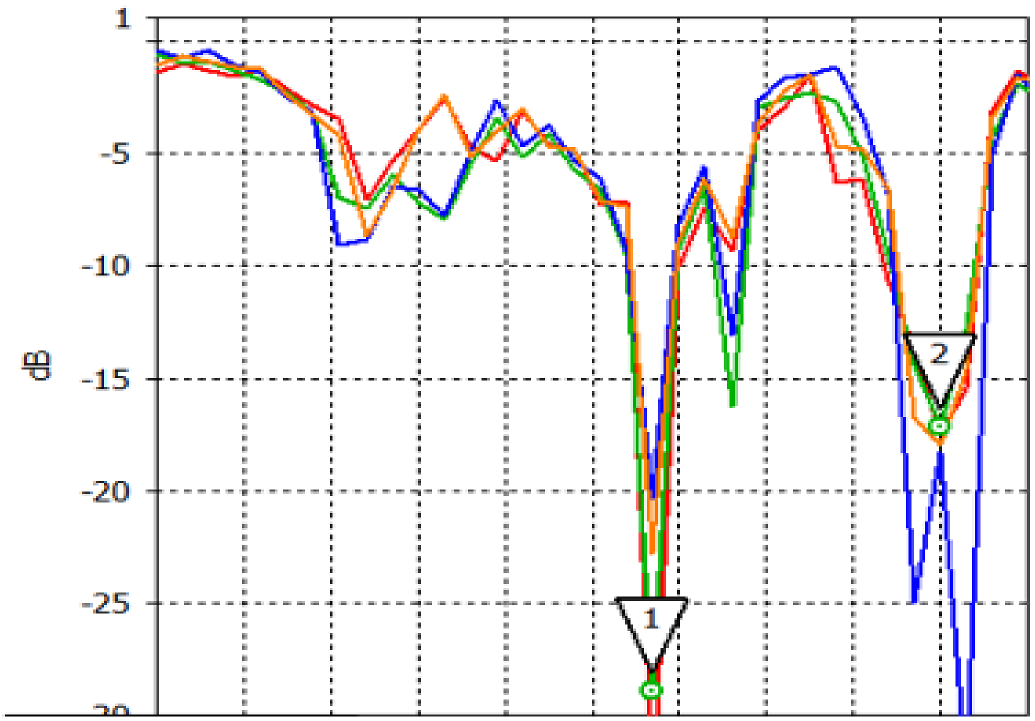
- **Configuration #1**
  - Upgrade of the initial design
  - Geometry keeps as much as possible the initial configuration
  - Processing of 8 tows will be possible



Examples of CEM for "Configuration 1". On this model, the tows are static.
In red are the locations of high field density.
These dynamic spots have a good covering of the tows.
Theoretically, the EM energy coupling on to the fiber tows looks very good.

Exploration of new configuration (Dec. 2019)

- **Configuration #1**
  - Example of tuning characteristics of the system (the CEM predicts the tuning characteristics of the generator, transmission line, and applicator)

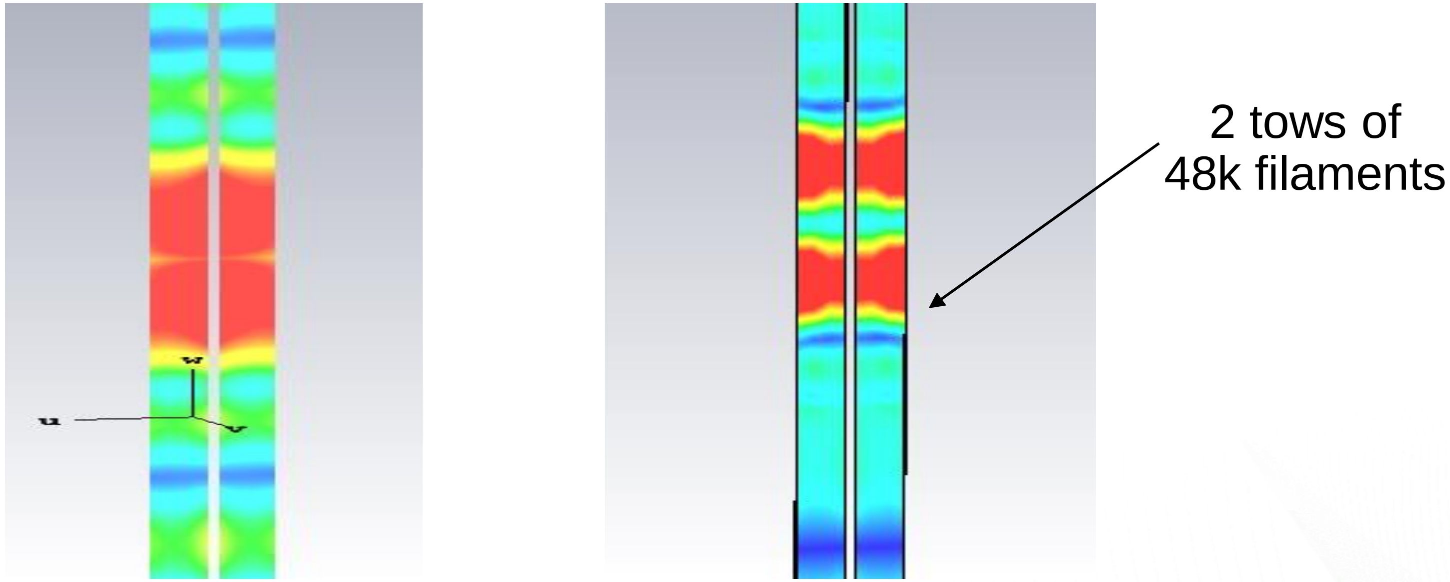


Examples of theoretical values (with CST computer model) of S11 over a band of interest.*
*Several configurations of the applicator with the load show favorable tuning opportunities**.*
For a setup such as CPEC-4, a good tuning is at -20dB or lower. Marker 1 indicates a narrow band with -29dB, which is ideal, whereas marker 2 shows a broader band with fair match at -17dB, which is still acceptable.

- *S_{11} : Coefficient of reflection: This is the indicator to measure the power reflected back to the generator.*
- *** This type of profile for S11 was validated experimentally with our initial hardware system.*

Model for new configurations (Dec. 2019)

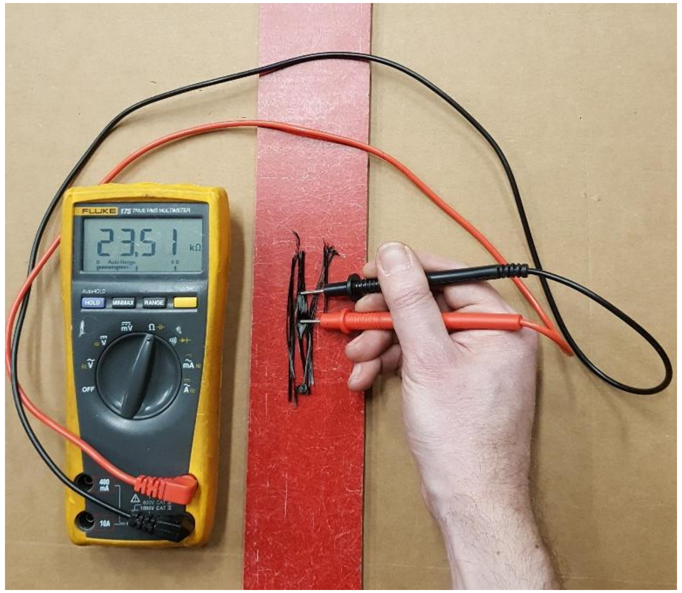
- **Configuration #2**
  - New geometry/configuration explored (attempt to reduce the constraints)
  - Focus of the distribution of the high field intensity on a pair of tows.
  - Process will be limited to 4 tows (up to 48k each)



The two pictures show two energy deposition patterns on pairs of tows with the second configuration modeled with the CEM (CST).
The difference between the two models' outputs represented here is due to tuning modification of the applicator.

First test with 2nd configuration (Apr. 2020)

- **Construction of the new configuration completed** on Apr. 27, 2020 (validation of milestone M13).
- Commissioning and preliminary test in progress (as of Apr. 27, 2020)
- First dry runs at low power with one tow showed evidence of carbonization.



Test #1: One static tow of 48k in air for 10min at low power. The fiber became conductive

Test #2: One static tow of 48k in N_2 for 10min at low power. The fiber clearly shows a color change, but no conductivity. The reason of this outcome is due to the settings of the processing parameters.

Note: the goal of the project is to carbonize 4 tows of 24k faster, and with performance equal to or better than conventional.

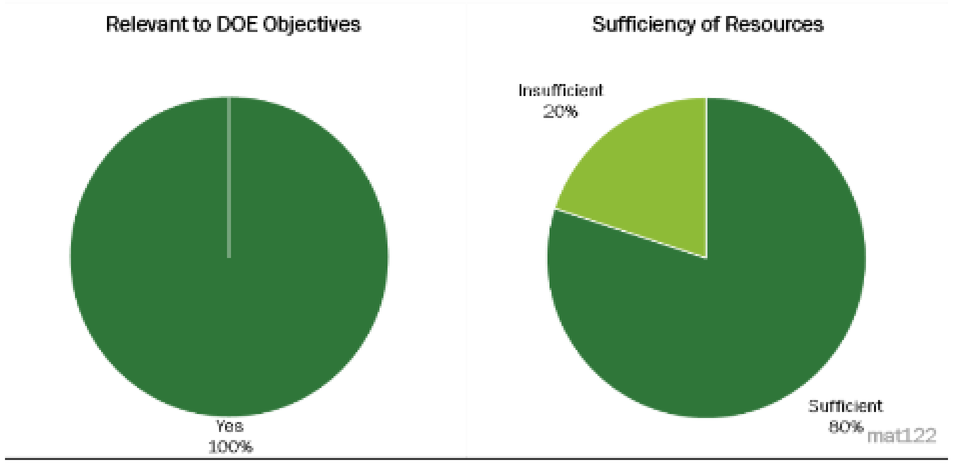
Generator non-conformity

- In February 2020, it was discovered that the generator had a non-conformity (compare to the initial technical specification/original PO):
  - Phase control existing but inappropriate
- This non-conformity is suspected to impact the operation of the setup and encouraged a dual approach:
  - Legal: ORNL indicated the impossibility of legal action against the manufacturer/retailer
  - Diplomatic: deal directly with the manufacturer's main technical supplier
- Negotiation began in early Feb. A technical solution was finally agreed upon in April.
  - Agreement of upgrading a part of the system
- Shipping is in process as of Apr 27, 2020.

As 4/27/2020

Response to Previous Year Reviewer's Comments

- Previous year scoring (AMR 2019):



- Question 1:

Approach to performing the work—the degree to which technical barriers are addressed, the project is well-designed, and well planned.

- Very positive comments from the five reviewers:
  - R #1: "The reviewer described the approach as outstanding. The project to scale up the technology is difficult…"
  - R #2: "The approach is creative…"
  - R #5: "… the approach… is both thoughtful and effective for reducing energy and improving throughput."; "… the scale up… is essential toward demonstrating the potential of reducing cost and expending capacity."

Our answer:

➢ Thank you for these favorable comments

Response to Previous Year Reviewer’s Comments

- Question 2:

Technical Accomplishments and Progress toward overall project goals—the degree to which progress has been made and plan is on schedule.

- R #2: “is addressing a significant technical challenge ranging from material development, equipment optimization, successive iterations, property optimization to meet DOE targets, and pathway to commercialization.”
- R #3:”CPEC-3 equipment was discussed and showed promising results”
- R #4: “progress is behind schedule due to supplier delivery and equipment difficulties. While this is disappointing, it is not uncommon.”
- R #5: “The reviewer could not find any computer modeling results to design the processing conditions”.

Our answer to reviewer #5:

➢ Some sanitized model can be public, but due to export control, no correlation can be presented between models and physical hardware, nor can pictures of the hardware can be disclosed.

Response to Previous Year Reviewer's Comments

- <u>Question 3:</u>

Collaboration and Coordination Across Project Team.

  - R #3: "key collaborator on this project as 4X Technologies and that relationship seems to be working well for the researchers."
  - R #4: "it is arguable that closer coordination between the stakeholders may have mitigated the delays and accelerated progress"
  - R #5: "there is no discussion in this presentation on the roles and responsibilities, or the interactions between ORNL and 4XTechnologies"

<u>Answer:</u>

- *To reviewer #4: Almost none of the required equipment is available off-the-shelf. Most of our devices either require customization to fit the need or design completely from scratch. This frequently takes longer than the lead times claimed by the suppliers. This was taken into account, especially regarding the purchase of the generator: it was ordered even before budget confirmation.*
- *To reviewer #4: It is very difficult to find company with the flexibility and the technical background (electromagnetic design, plasma technologies and material processing, especially fiber processing). Geographically, 4XTechnologies is in close proximity to ORNL.*
- *To Reviewer #5: The responsibilities are well defined in the contract. ORNL is in charge of the budget, HTC conversion, fiber evaluation, project evaluation, and technical support. 4XTechnologies is responsible for the modeling. Design and construction is a cooperative effort.*

Response to Previous Year Reviewer's Comments

- Question 4:

Proposed Future Research—the degree to which the project has effectively planned its future work in a logical manner by incorporating appropriate decision points, considering barriers to the realization of the proposed technology and, when sensible, mitigating risk by providing alternate development pathways.

  - R #2: "Future work is very challenging, but a good plan is in place" *(we agree, this is R&D work has been very challenging !)*
  - R #3: "Project part of a comprehensive solution for full carbonization process based on CPEC technologies (…) is relevant and worthwhile direction for future research"

Answer:

➢ *Thank you for these encouraging comments.*

- Question 5:

Relevance—Does this project support the overall DOE objectives?

  - R #1: "… the project is highly relevant to the DoE objectives and addresses the immediate need of industry…"
  - R #2: "… addresses the urgent need of LCCF for lightweight vehicles."
  - R #4: "… this project has high DoE relevance because it is directly because it is addressing directly ways of reducing the energy costs of producing CF."

Answer:

➢ *We thank the reviewers for the positive perception of this project.*

Response to Previous Year Reviewer's Comments

- <u>Question 6:</u>

Resources—How sufficient are the resources for the project to achieve the stated milestones in a timely fashion?

  - "… the team has good resources through the large assets at ORNL in the CF space, and supporting technologies at 4XTechnologies".
  - "A very reasonable level of funding early stage research work was observed by this reviewer. The capital-intensive component of this research is an important cost to bear."
  - "This reviewer indicated that the resources are likely to be insufficient, and explained that delays in equipment delivery and commissioning will undoubtedly will require additional funding to complete this important project."

<u>Answer:</u>

➢ *Due to technical issues, the project time schedule could not be maintained. As a consequence, the budget was reevaluated in Oct. 2019. At this moment, it seems that the funding will be appropriate.*

Collaboration and coordination

ORNL performed this project in collaboration with:

4XTechnologies — Joint development. Equipment construction and experimental work performed at this site.

4XTechnologies is a dynamic startup located in Knoxville, TN, with a core focus on plasma science and engineering and experience in fiber treatment/conversion and environmental applications.

Remaining Challenges and Barriers

- Process material and achieve DoE's programmatic required mechanical properties with the new configuration.
- Complete the modifications into initial configuration. This will proceed in parallel with the fiber processing in configuration #2.
- Execute generator upgrade with an acceptable lead time (less than a month) to test with all configurations.
- Ensure proper full-scale operation of CPEC-4 as predicted with acceptable uniformity across the width of the tow band.

Proposed Future Research

- FY20
  - Upcoming work:
    - Fulfillment of M14-M15: Normal operation of CPEC-4 with 4 tows 24k with 60s residence time, achieving 250 ksi/25 Msi.
    - An economical evaluation of the technology per MS16.
  - This project is scheduled to come to conclusion at the end of FY2020.
  - Propose research for a comprehensive solution for full carbonization process based on CPEC technology.

Any proposed future work is subject to change based on funding levels

Summary

- CPEC-4 processing system was:
  - Tested
  - Altered
  - Showed evidence of carbonization
- Project was extended for one more year.
- Significant issue remains with the generator system:
  - Inadequate phase control performance has to be addressed by the technical supplier.
- One new processing configuration is complete and ready for testing (Apr. 27, 2020).


Mat122
Questions?
Thank you for your attention

Technical Backup

Timeline/delay justification, Power generator issue

The generator needed for this project is a new product with features that have not yet been available on the market. Unfortunately, development of these features by the manufacturer led to significant delays in delivery as well as performance that did not meet the criteria initially promised.

Date of action	Expected delivery time at 4XTechnologies	Comments
2/21/2018	7/15/2018	Original commitement (purchase order issued by ORNL)
6/15/2018	8/15/2018	Manufacturer requests more time for testing: Reengineering of the back panel needed
8/7/2018	9/24/2018	Procuremnet issue
9/26/2018	10/15/2018	Manufactureing issue with a smaller version
1/8/2019	2/15/2019	Teleconference between ORNL, 4XT, and the manufacturer: commitment to ship the system to a partner/contractor to complete the construction
1/28/2019	N/A	Reception of the system by the partner: Beginning of evaluation of the work
1/30/2019	3/8/2019	Teleconference between ORNL, 4XT, and the partner: Remaining work-load estimated: ~10%
3/12/2019		First part of the system delivered (2nd part expected by the end of May)
6/15/2019		Second part of the system delivered
2/14/2020		**Issue with the phase control identified. Search for a technical solution with the technical supplier**
4/16/2020		**Technical solution identified: Need to return part of the system to the technical supplier for 3 weeks**
4/30/2020		**Shipping date of a part of the system**

Technical Accomplishments (FY2018)

Continuous Processing of Fiber with CPEC-3 Furnace

Mechanical properties of fully carbonized fiber (as of 11/2017)

Oxidation (conventional), LTC (CPEC-3), HTC (Conventional)*

Test#	Density (g/cc)	Diameter (Avg) µm	Std. Deviation	Tensile Strength (Avg) ksi	Std. Deviation	Modulus (Avg) Msi	Std. Deviation	Strain (Avg) %	Std. Deviation	Residence Time
1	1.8032	8.05	0.35	348.70	77.50	23.42	1.84	1.49	0.28	Long
2	N/A	8.20	0.41	303.00	87.50	22.73	2.76	1.40	0.32	Short
2	1.7924	8.44	0.74	356.60	135.30	24.88	3.83	1.42	0.47	Long
2	N/A	8.00	0.80	254.20	88.90	21.42	2.59	1.22	0.43	Long
3	N/A	8.40	0.53	333.00	149.80	25.44	3.45	1.29	0.51	Short
3	N/A	8.22	0.63	292.00	91.70	22.79	3.31	1.27	0.27	Short
3	N/A	8.42	0.46	331.30	125.00	23.44	1.84	1.48	0.55	Long
4	N/A	8.09	0.62	354.60	97.60	23.64	2.42	1.48	0.32	Short
4	N/A	8.06	0.72	263.60	132.80	22.31	3.61	1.13	0.44	Short
4	1.8138	8.91	0.63	340.20	101.70	25.14	1.73	1.39	0.43	Long
4	1.8135	8.73	0.56	285.50	98.50	23.07	2.03	1.23	0.37	Long

Table 1: Mechanical properties of fully carbonized samples at HTC. All residence times in CPEC-3 are shorter than 90 seconds. The values highlighted in green surpassed the dual programmatic requirements of 250ksi tensile and 25Msi modulus simultaneously.*

* HTC: High Temperature Carbonization

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